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Preparation of a New Heavy Metal Chelating Agent and Its Removal of Copper Ions in Wastewater

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ABSTRACT

In this study, a new type of dithiocarbamate (DTCs) heavy metal chelating agent MF-DTC was prepared by carbon disulfide modified melamine formaldehyde resin prepolymer, and the structure of the chelating agent and chelating products was characterized by infrared, ultraviolet, elemental analysis, thermogravimetry, scanning electron microscopy and other means: the process of chelating agent Cu was studied, and the results showed that MF-DTC chelating agent had a higher adsorption capacity, and compared with the traditional chelating agent SDD and Na_2S, it had higher chelating capacity under acidic conditions, it can be directly used to treat heavy metals in acidic wastewater. Moreover, the environmental stability of heavy metals after chelation is high. Heavy metals are not easy to be leached again after treatment.

KEYWORDS

Melamine Formaldehyde resin; Dithiocarbamate; Cu (II); Chelating agent

1. INTRODUCTION

Heavy metal pollution is mainly derived from the waste incineration fly ash, plating, metal smelting and other industries. Due to heavy metal difficulty biodegradation and high toxicity, if it is directly discharged into the environment without treatment, it will be enriched in the human body and the environment. Ecological system and human health [1-4]. The current heavy metal treatment methods mainly include membrane separation method, bioccoencies, adsorption method, ion exchange method, and chemical precipitation method [5, 6]. Compared with other treatment methods, the chemical precipitation method has become the main method of heavy metal treatment [7, 8]. in recent years due to its simple operation, low cost, and safety and environmental protection [9, 10].

The chemical precipitation method is a compositional effect of heavy metals and chelating agents, and it generates chelars that are not easy to migrate with small solubility accumulation, thereby achieving the method of separating heavy metals [11-13]. Siululous carboxyl-carboxyl acid salt (DTC) class is a nuclear bonus reaction with a polymer or monomer containing amino group under alkaline conditions in alkaline conditions; The group can chelate heavy metals in the form of covalent and controlled keys. Organic sulfur (S) can form a two -dimensional or three -dimensional mesh structure with heavy metals [14, 15]. The influence has more studies in the heavy metal treatment industry [16, 17]. However, it is more in experimental research. Due to the expensive raw materials, the migration of heavy metals, difficulty in operating, and unstable under acidic conditions, its industrial application is limited. Therefore, the low -priced, stable and efficient chelator has good application prospects [7, 18, 19].

Tsichalamine formaldehyde resin (MF) is one of the earlier industrialized polymer chemical products. Its raw materials are widely sources and are mature in synthesis. Medicine and other industries [20-22]. Due to the stable six-membered ring structure and a large number of amino groups, it provides a prerequisite for the preparation of DTCs chelating agent. Therefore, This study synthesizes the intermediate through the modified melamine formaldehyde resin, and the insoluble melamine formaldehyde resin is formed by adding hydroxyl -based melamine cross -linking synonyms by adding hydroxyl menia. The salt group can quickly form a stable and insoluble substance with heavy metal ions. The price is cheap, and it can be produced in large -scale industries to deal with the problem of heavy metal pollution in flying ash and industrial wastewater.

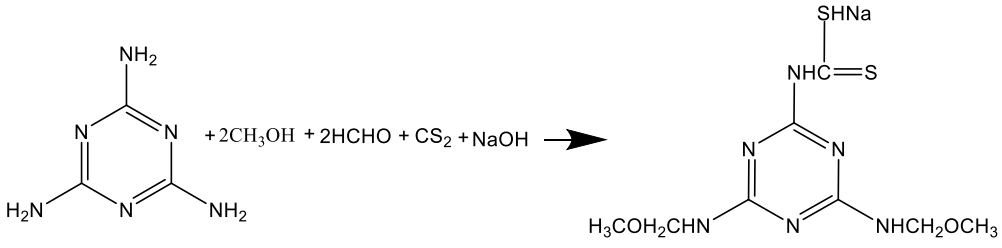
2. EXPERIMENTAL

2.1. Materials and Methods

Melamine (M), formaldehyde (F), methanol, carbonide (CS_2), sodium hydroxide were used as received without purification. Solvents and other chemicals used were of analytical grade. Elemental analysis was done using a Perkin-Elmer Elemental Analyzer series II Model 2400. FT-IR spectra recorded on a Perkin Elmer 16F PC FTIR spectrometer. The microscopic morphology was observed by HITACHI-SU8020 scanning electron microscope. The thermal gravimetric analysis of chelating agent and metal chelate was carried out by American TA-DSC25 thermal gravimetric analyzer. The test was carried out in a nitrogen atmosphere. The full-spectrum scanning of MF-DTC was performed using a V / UV-1500 C ultraviolet spectrophotometer produced by Shanghai Meixian Instrument Co.Ltd. The concentration of Cu^{2+} in the solution was measured by V/UV-1500C ultraviolet spectrophotometer produced by Shanghai Meixi Instrument Co. Ltd. The concentration of Cu^{2+} in the solution was measured by V / UV-1500C ultraviolet spectrophotometer produced by Shanghai Meixi Instrument Co. Ltd.

2.2. Preparation of MF-DTC

Melamine (0.12 mol) was prepared into an aqueous solution of 10 wt. %, and then formaldehyde solution (0.24 mol) and anhydrous methanol (0.24 mol) were added ; after that, the pH was adjusted to 11 with NaOH and stirred in a 60 ℃ water bath for 1 h. After cooling to room temperature, sodium hydroxide (0.15mol) and carbon disulfide (0.18mol) were added, and the brown yellow aqueous solution was obtained after stirring at 600 r / min for a certain time. The solid product MF-DTC can be obtained by freeze-drying for 24 h. The Scheme 1 shows the reaction equation.



Scheme 1. Preparation principle of MF-DTC chelating agent

2.3. Adsorption Experiments

The precipitate obtained from the treatment of Cu^{2+} wastewater by chelating agent was washed three times with deionized water and dried in a vacuum drying oven. The toxicity of precipitation leaching was tested according to the “acetic acid buffer solution method for leaching toxicity of solid waste leaching method (HJ/T 300-2007)”. During the test, the pH of acetic acid solution was 2.64, and the solid-liquid ratio was maintained at 1: 20. Compared with the commonly used chelating agents such

as sodium thiram and sodium sulfide, the supernatant was taken at regular intervals to measure the concentration of Cu^{2+}.

HCl and NaOH solutions were used to adjust the pH values of pure water to 1, 3, 5, 7 and 9, respectively, to simulate different acid-base environments. A total of 0.1 g metal chelates were added to 100 mL of different pH solutions, mixed evenly and allowed to stand. After each period of time, 1 mL of supernatant was taken and filtered through a 0.45 μm filter membrane. The residual Cu^{2+} concentration was measured using an ultraviolet spectrophotometer, and the Cu^{2+} leaching concentration was calculated.

3. RESULT ANALYSIS AND DISCUSSION

3.1. Catalyst Characterization Result

3.1.1. UV full spectrum analysis.

The MF-DTC solution UV full-spectrum scanning is shown in Figure 1. MF-DTC has a maximum absorption peak at 250 nm and 285 nm. 250 nm is N-C = S Group π-π*jump, and the absorption peak near 285 nm is the peak of 285 nm. S-C = N-swarm of N-π*jumping to the anti-key π orbit in the S group [23]. Therefore, it shows that MF-DTC contains dimer sulfur amino groups (-NCSS-).

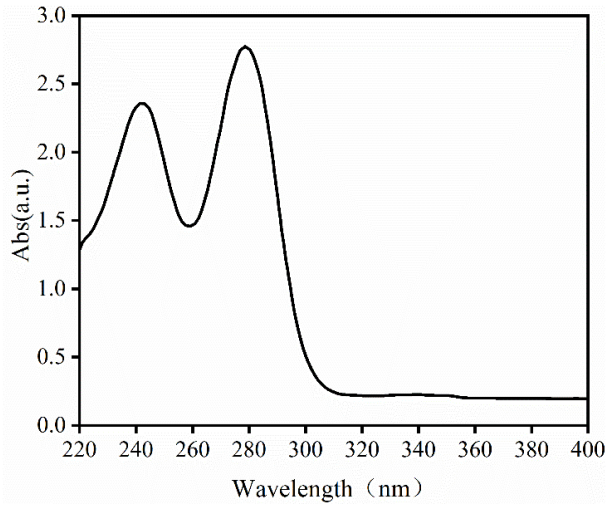


Figure 1. The UV-vis pattern of MF-DTC

3.1.2. FT-IR analysis.

Hydroxymeric melamine premature MF-OH, chelated MF-DTC metal chelated MF-Cu infrared appearance results are shown in Figure 2. From Figure 2a and 2b, it can be seen that MF-DTC is at 1360 cm^{-1} for C=S telescopic vibration peak, 810cm^{-1} and 590 cm^{-1} is the C-S key telescopic vibration peak in the DTC group, indicating that in the MF-DTC chelating agent Muslide was successfully introduced into a mixture (-NCSS-). When MF-DTC and Cu^{2+}chelated, the C=S telescopic vibration peak moved from 1360 $cm^{-1}$to 1316 cm^{-1}; the C-S key telescopic peak at 810 cm^{-1} and 590 cm^{-1} disappeared Show, it shows that in the chelation process, the supporting role of S atoms and Cu^{2+}in the DTC group [24, 25].

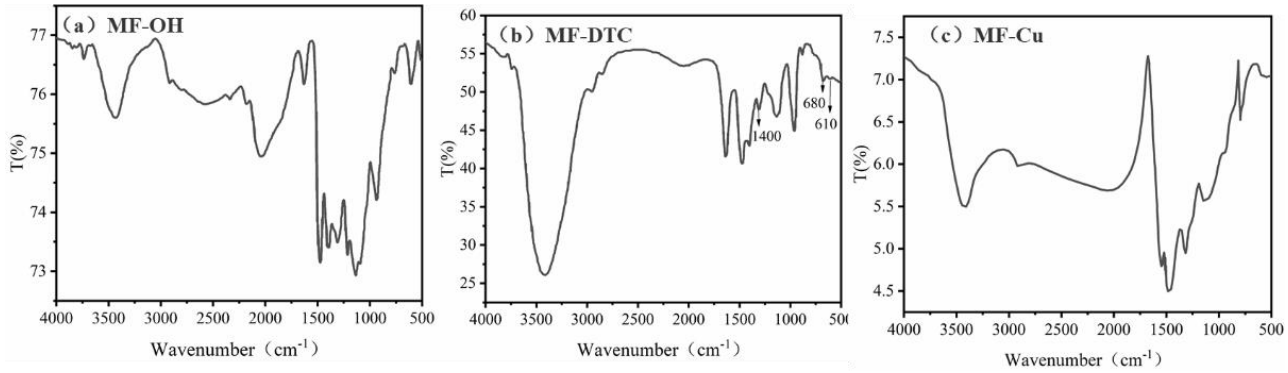


Figure 2. The FTIR analysis of MF-OH, MF-DTC and MF-Cu

3.1.3. Elementary analysis.

The results of elemental analysis of MF-DTC are shown in Table 1. The molar ratio of N/C in MF-DTC is 1:1.02, while the theoretical N/C ratio in melamine is 2:1, indicating that after the modification of melamine by formaldehyde, the N/C ratio decreases due to the introduction of formaldehyde, and the magnitude of the decrease proves that CH_2O: MF is close to 2:1 reaction, which is close to the feed ratio of the experimental process. At the same time, 13.16 % S element was added to MF-DTC, indicating that CS_2 was successfully added to hydroxymethyl melamine. The molar ratio of N / S-S in MF-DTC is 5.25, which is higher than the theoretical value of 3:1. It may be due to the condensation reaction in hydroxymethyl melamine to form oligomeric hydroxymethyl melamine.

Table 1. Element analysis of MF-DTC

Chelating agent	element content (%)				N/C	N/S-S
	C	N	H	S		
MF-DTC	25.32	30.24	4.222	13.16	1.02	5.25

3.1.4. TG analysis.

Figure 3 is TG and DTC analysis results of M, MF-DTC and MF-Cu. MF-DTC, and MF-Cu's weight loss rates were reduced by 99%, 84%, and 52%, respectively, indicating that the melamine was successfully introduced into other elements after modification, and the heavy metal in the MF-Cu was successfully checked. The raw material MF has only one loss stage, mainly 280 ~ 380 ℃, which is the thermal solution process of melamine molecules. The MF-DTC and MF-Cu both have three weightless stages. The first stage is 100 ℃ ~ 126 ℃, mainly due to the volatilization of the crystal water absorption of chelated agent and the crystallization of metal chelates; the second stage is 170 ℃ ~ 220 ℃, mainly the decomposition process of sulfur carboxyl group; the third stage mainly occurs at 330 ℃ ~ 380 ℃, mainly the thermal solution process of melamine. At the same time, the disintegration temperature of sulfurization in metal chelates is 170 ℃, which is much higher than the ambient temperature, indicating that the metal chelates and chelating agents have good thermal stability.

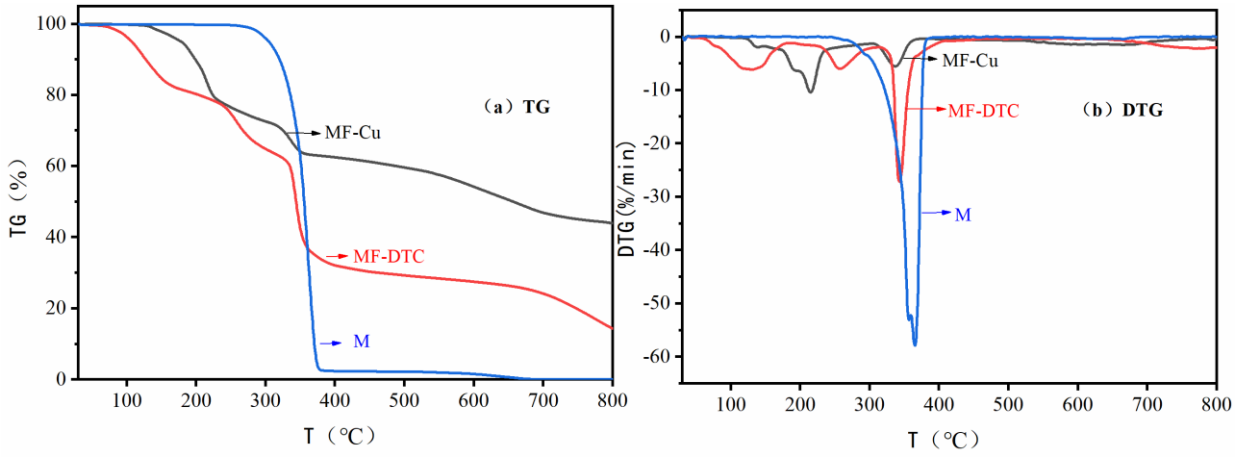


Figure 3. Comparison of the thermal stability of MF-DTC and MF-Cu

3.1.5. SEM analysis.

The analysis of the MF-DTC and MF-Cu scanning electron microscopic electron microscopic electron microscopic electron microscopic electron microscopic electron microscopic is shown in Figure 4. Figure 4a is a MF-DTC chelating agent, showing a mesh structure with small particles stacked, and there are more gaps in the middle, which is conducive to heavy metal adsorption. Metal chelates (Figure 4b) is a block-shaped particles with uneven size, and the appearance of block particles. Firming is conducive to the stability of metal chelates, indicating that during the chelating process, heavy metals may be fixed by roll sweeping and parcels [26].

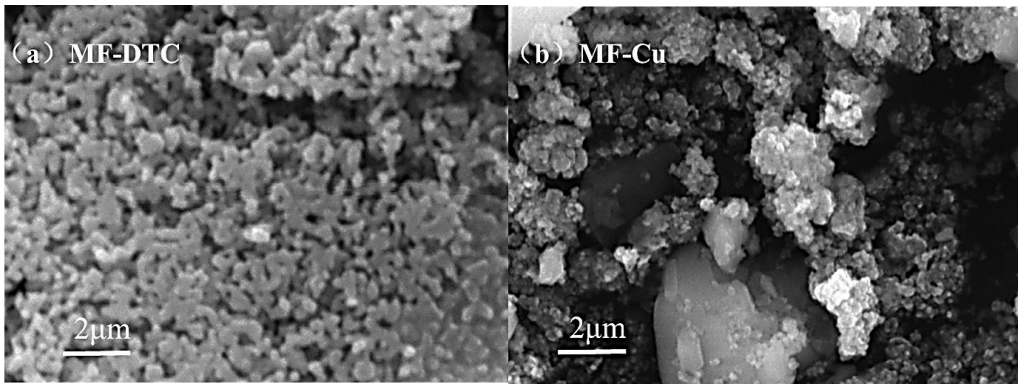


Figure 4. SEM image of MF-DTC and MF-Cu

3.2. Initial pH Effect of Solution

The heavy metal wastewater is mostly acidic wastewater, which is to explore the applicability of chelating agents in handling wastewater. HCl and NaOH are used to adjust the Cu^{2+}solution pH to 1 ~ 9, and then add the same mass MF-DTC, SDD and Na_2S chelating agent. Stir for 20 min to rest for 10 min to measure the Cu^{2+}concentration of the supernatant. The experimental results are shown in Figure 5: It can be seen from the figure that compared with the commercial chelating agent SDD and Na_2S, MF-DTC shows better chelating performance under strong acidic conditions. At PH = 1, the MF-DTC removal rate of $Cu^{2+}$is higher than 90%, while SDD and Na_2S have only 62.3%and 10.7%of the Cu^{2+}removal rate; at PH> 3 95%, it is proved that the DTC chelating agent has a high extent effect under acidic conditions compared to inorganic chelating agents; at the same time, it shows that MF-DTC chelating agent has a higher removal effect under acidic conditions compared to commercial DTC chelating agent Fumi sodium. Essence In addition, under the same amount, as the pH increases, the removal rate has gradually risen. This is due to the high H^+concentration under strong acidity conditions, which will be chelated on the chelating agent with heavy metal competitions to reduce chelars Hyposimal chelating performance. Cu^{2+}has gradually occurred at pH> 6, so the effect of Cu

$(OH)_2$ can be excluded. To sum up, MF-DTC has a high removal rate under acidic conditions, which can be directly used to deal with acidic heavy metal wastewater, reflecting good application prospects.

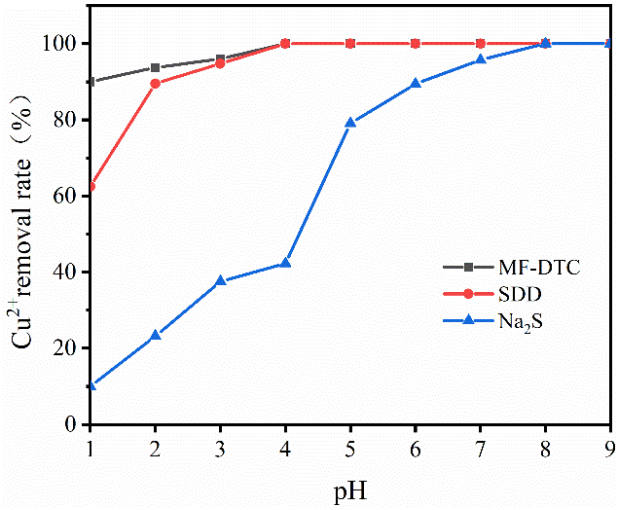


Figure 5. Removal of Cu by chelating agents at different pH

3.3. Metal Chelated Stability

Heavy metal chelates are affected by environmental pH, and there is a risk of secondary immersion. Therefore, this article refers to the "Solid waste immersion toxic immersion method (HJ/T 300-2007)" to test the toxicity of the precipitation. As shown in Figure 6b. Compared with the inorganic chelating agent Na_2S, MF-Cu and SDD-Cu are high in stability when soaking in acetic acid buffer solutions. The chelating products of SDD and MF are basically not analyzed after soaking for 10 h in the acetic acid buffer, which is in line with national hazardous waste. Detection standards, low toxicity.

It can be seen from Figure.6a that the leaching concentrations of MF-Cu at pH = 1 and 3 for 2~32 h were 6.60 mg/L and 2.13 mg/L, respectively, indicating that MF-Cu had high stability under strong acidic conditions. When the pH is greater than 3, the leaching concentration of MF-Cu is less than 1 mg/L within 2~32 h, and the leaching fraction is less than 0.06 %, indicating that MF-DTC is less affected by environmental factors after chelating heavy metals, and the risk of secondary leaching of heavy metals is low.

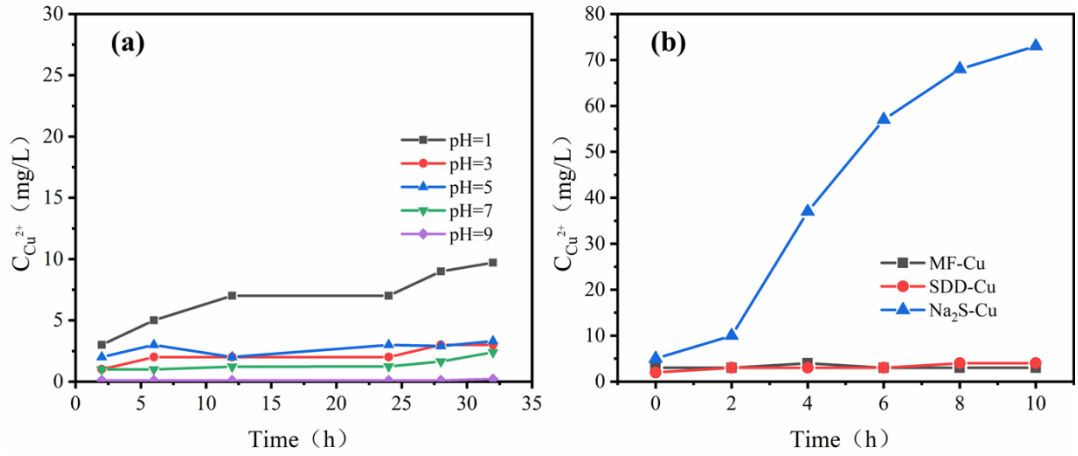


Figure 6. Acid and alkali resistance of MF-Cu

3.4. Adsorption Thermodynamics

3.4.1. The temperature attachment curve at different temperatures.

At 30 ℃, 40 ℃ and 50 ℃, 8.3 mg MF-DTC chelating agents were added to 100 mg/L, 200 mg/L, 300 mg/L, 400 mg/L, and 500 mg/L. After the stirring reaction of 600 r/min for 4 h, set the solution for 10 min, take the solution to measure the Cu^{2+}concentration, and calculate the balance with the initial concentration of the balance of the balanced adsorption at different temperatures. The experimental results are shown in Figure 7.As can be seen from the figure: At the same temperature, the balanced adsorption volume gradually increases with the increase of the initial concentration of Cu^{2+}, indicating that a single chelator molecule can match multiple Cu^{2+}; when the initial concentration of Cu^{2+} Gradually increased, indicating that the chelating process is the process of heat absorption, and the increase in temperature is beneficial to the chelating process.

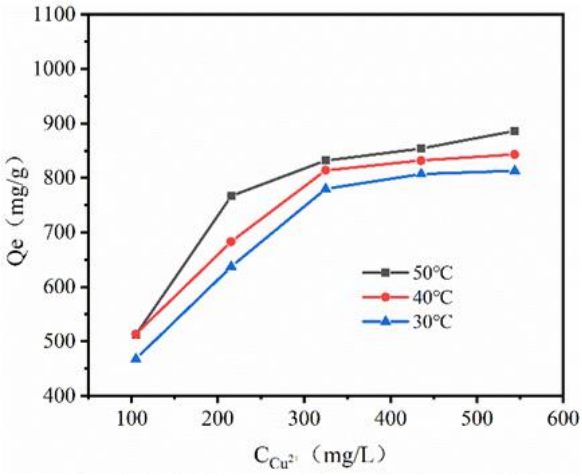


Figure 7. Isothermal adsorption line

3.4.2. Waiting temperature adsorption model

The adsorption isotherm model refers to the concentration relationship curve between solute molecules when the adsorption on the two-phase interface reaches equilibrium at a certain temperature. The Langmuir isotherm represents the reversible adsorption of a monolayer, and the equation is [27]:

$$\frac{C_e}{q_e} = \frac{1}{q_m K_l} + \frac{C_e}{q_m} \tag{1}$$

In the formula: q_m saturated adsorption, mg/g; k_l is adsorption balance constant. With C_e and C_e/q_e as horizontal and vertical coordinates, the Langmuir adsorption is the temperature line diagram, and k_l and q_m can be calculated based on the slope and interception of linear fitting.Freundlich equivalent equation equation expression is as follows[27]:

$$ln\ q_e = ln\ K_f + \frac{1}{n} ln C_e \tag{2}$$

In the formula: K_f is the Freundich adsorption coefficient; n is the Freundich constant. Taking $\ln C_e$ as the horizontal coordinate and $\ln q_e$ vertical coordinates, the Freundich adsorbing the equivalent is used to calculate K_f and n based on the slope and intercept of linear fitting.

At 30 ℃, the temperature equations such as Langmuir and Freundlich are used to fit the MF-DTC chelating Cu^{2+}experimental data. The experimental results are shown in Figure 8 and Table 2.

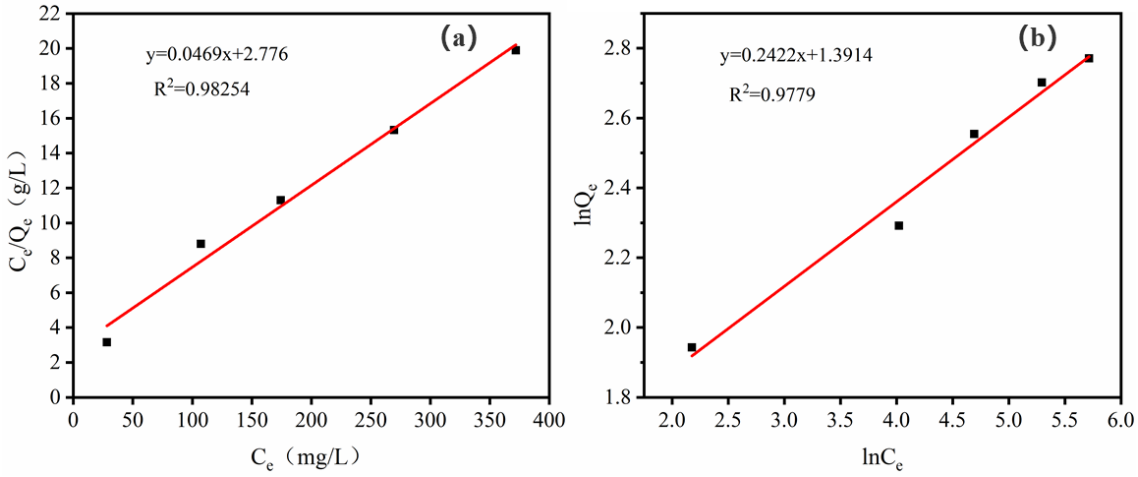


Figure 8. (a) Langmuir isotherm model fitting on the adsorption of Cu^{2+} ions. (b) Freundlich isotherm model fitting on the adsorption of Cu^{2+} ions

It can be seen that the LangMuir model of the MF-DTC chelated copper ion process is higher than that of the temperature adsorption model such as Freundlich.

Table 2. Adsorption isotherm models of Cu^{2+} by MF-DTC

Langmuir	K_l(L/mg)	0.576
	q_m(mg/g)	647.32
	R^2	0.9825
Freundlich	K_f(mg/g)	467.32
	n	5.43
	R^2	0.9779

3.4.3. Calculation of thermal parameters.

In order to explore the temperature effect during the Cu^{2+} process, thermodynamic calculation was performed, and Gibbes Freedom can change ΔG, ΔH, and entropy variable ΔS according to the formula calculation of the adsorption response.

$$\Delta G = -RTlnK_l \tag{3}$$

$$\ln K_l = -\frac{\Delta H}{RT} + \frac{\Delta S}{R} \tag{4}$$

$$\Delta S = (\Delta H - \Delta G)T \tag{5}$$

In the formula R is thermodynamic constant, 8.314J/(mol.k); ΔG is Gibbes freedom; ΔH is Enthalpy changes, KJ/mol; ΔS is Entropy changes, $J \cdot mol^{-1} \cdot k^{-1}$. The calculation results are shown in Table 3:

Table 3. Thermodynamic data and activation energy for Cu^{2+} ions adsorption on MF-DTC.

T/K	ΔG (kJ/mol)	ΔH (kJ/mol)	ΔS ($J \cdot mol^{-1} \cdot K^{-1}$)
303.15	-8.84	12.4563	32.3765

It can be seen from the table, ΔG <0, chelating reaction spontaneously. ΔH> 0, chelating adsorption to heat-absorbing reaction, heating can increase the reaction, confirming that the temperature adsorption model at different temperatures concluded that the heating reaction is beneficial to chelating reactions.

4. SUMMARY

In this study, the DTC chelating agent MF-DTC was prepared by modifying the low-cost melamine formaldehyde resin prepolymer. It was found that MF-DTC contained a stable six-membered ring structure and DTC group by infrared spectroscopy and ultraviolet spectroscopy, and the product analysis after chelation showed that the DTC group formed a coordination bond with heavy metals ; elemental analysis showed that the S content in MF-DTC was 13.0 %. The saturated adsorption capacity of the optimal chelating agent for 200 mg/L Cu^{2+} was 641 mg/g. Compared with sodium formate and Na_2S, it has better chelating performance, and has higher removal effect on Cu^{2+} in a wide pH range (3~9), and the risk of secondary leaching of heavy metals is low, which meets the requirements of national hazardous waste treatment. The study of chelating thermodynamics shows that the process of MF-DTC chelating Cu^{2+} conforms to the Langmuir isothermal adsorption model, and it is a spontaneous and endothermic process.

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